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COMMENTS ON THE HANDLING OF PLUTONIUM

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I. INTRODUCTION

A. General

Prompted by increased emphasis on research involving plutonium, the following outline of problems, procedures, techniques and approaches generally applicable to laboratory-scale handling of plutonium and its compounds has been assembled. It is the purpose of this review to indicate the broad scope of past experience in this field, to discuss some of the techniques and the philosophies currently in use and to outline some guiding thoughts applicable to future expansions.^a Finally, the review may offer the novice a general picture of past and present developments as a prelude to experimental work in this field. For more detailed information than that presented here, the references cited should be consulted.

A discussion of this sort can neither be completely authoritative nor all inclusive. In order to make this review useful for the purposes mentioned, the discussion of many topics has had to be severely limited. Many details available in the references have not been included. An attempt has been made to avoid a dogmatic approach to some of the more fundamental discussions. Because of these limitations, the lack of data specifically applicable to a particular problem or research program and the unavoidable omission of references to some of the published literature, this review should be regarded only as advisory. A number of points need to be established at the outset. Normally, a laboratory facility in which plutonium is being handled has been designed for use with radioactive materials. There exist, therefore, procedures for periodical medical examinations, routine blood and urine counts, survey schedules, emergency procedures,^(3,48) etc. It is also not necessary to spell out in detail physical features of a plutonium laboratory which, at least in rudimentary form, are likely to be already present. Features falling in this class include delineation of rooms according to risk,^b the use of personnel

^a Broad reviews of this type for plutonium are not available. See, however, reference 10 for a complete description of an older and lower level facility, and reference 11 for a review of uranium handling. A historical survey is given in references 18 and 45. Several symposia on the subject have been held by the British (see reference 47).

^b Isolation rooms, red areas, purple areas, high and low level areas, etc. Nomenclature varies considerably among the sites but in principle, (isolation) all of these designations are identical.

protection devices such as dosimeters, the details of protective clothing normally used, regulations pertaining to disposal of aqueous waste and the use of drains, etc. It must be assumed that all such procedures are functioning in facilities which handle plutonium. Finally, references to detailed procedures (emergency or fire, for example) developed at specific sites are normally not directly applicable elsewhere^a and will only be referred to in the text and bibliography.^b

B. Quantitative Aspects of the Plutonium Hazard

The quantitative aspects of the hazards associated with plutonium have been discussed in the literature.^c It was recognized early that soluble plutonium, like many other heavy metal elements, concentrates in the bone. The critical organ for insoluble plutonium is the lung. The short range alpha particles cause severe local destruction of tissue irrespective of the location. In addition, plutonium has a long biological half-life.^d These features make plutonium a severe poison. In order to appreciate more fully the severity of the hazards, the current maximum concentration limits applicable to several circumstances are listed in Tables 1 and 2. The inclusion of data on isotopes of plutonium other than Pu²³⁹ serves to emphasize the confinement problems which may arise as the source of plutonium shifts from production to power reactors.^e It is clear from the data shown in Table 2 that the permitted concentration for laboratory air, for example, precludes the use of visual or even crude radiation detection of contamination levels deemed safe for continuous exposure. This fact, perhaps more than any other, influences the methods of handling applied to particular plutonium samples.

In addition to the hazard due to inhalation, ingestion and the puncture mode of entry of plutonium, several other factors must be mentioned. Some of the work involving plutonium also involves intimate mixtures of plutonium and certain light elements. Due to (α ,n) reactions of these light elements, a considerable neutron flux may be associated with

^aThe use of the term "site" is meant to include all activities involving plutonium at a particular location. Such assignment should be adequate for this review. The sites at which plutonium work is in progress in the U.S. are ANL, LASL, RFP, UCRL (Berkeley and Livermore), ORNL, BMI, MIM, and HW.

^bDiscussions on parts of the area covered in this review may be found in references, 4, 9, 10, 11, 12, 15, 23, 24, 32, and 47.

^cSee for example reference 10b, TID-1002 and reference 3.

^dApproximately 20% is excreted in 50 years.

^eHeavy isotope build-up and concentrations likely to be found in power reactors fuels have been discussed elsewhere. See for example ORNL-2126 and HW-48078. See also reference 17.

Table 1

DECAY CONSTANTS AND CHARACTERISTIC RADIATIONS
OF SEVERAL PLUTONIUM ISOTOPES

Mass	Half Life	Energies in Mev			Activity ^a g/curie
		Alpha	Beta	Gamma	
238	86.4 y	5.5; 5.45	-	0.04	5.75×10^{-2}
239	24360 y	5.15; 5.13; 5.10	-	0.05	16.2
240	6580 y	5.16; 5.12	-	0.04	4.5
241	13.0 y	-	0.02	-	8.7×10^{-3}
242	2.79×10^5 y	4.90; 4.86	-	-	255

^a Approximate

Table 2

PERMISSIBLE CONCENTRATIONS FOR PLUTONIUM

Mass	Water ^a ($\mu\text{g/ml}$)	Air ^{b,d} ($\mu\text{g/ml}$)	Maximum Body Level ^c (μg)
238	8.7×10^{-8}	1.1×10^{-13}	4.6×10^{-4}
239	2.4×10^{-5}	3.2×10^{-11}	0.13
240	6.7×10^{-6}	9×10^{-12}	3.6×10^{-2}
241	1.3×10^{-8}	1.7×10^{-14}	7×10^{-5}
242	3.8×10^{-4}	5.1×10^{-10}	2

^a Based on $1.5 \times 10^{-6} \mu\text{c/ml}$ for continuous exposure (reference 3)

^b Based on $2 \times 10^{-12} \mu\text{c/ml}$ for continuous exposure (reference 3)

^c Based on insoluble Pu $8 \times 10^{-3} \mu\text{c}$; soluble Pu limit is $4 \times 10^{-2} \mu\text{c}$, (reference 3)

^d Corresponding value for uranium is $\sim 5 \times 10^{-5} \mu\text{g/ml}$.

such samples. For example, samples of plutonium fluorides can be calculated to emit fast neutrons at an approximate rate of $23,000 \text{ n}/(\text{sec})(\text{g Pu}^{239})$ from the interaction of the decay alpha particles and fluorine atoms.^a Samples of plutonium containing appreciable quantities of Pu^{238} , Pu^{240} , and Pu^{242} will emit fast neutrons from the spontaneous fission of these isotopes.⁽¹⁷⁾ This source of neutrons is not dependent on the chemical state of plutonium but only on the isotopic composition. Several of the plutonium isotopes emit weak gammas as part of the decay. These gammas, although not very energetic, contribute to the radiobiological hazard and can be ignored only in those cases where protection against the beta-gamma radiation from fission products is included in the manipulatory equipment.^b Finally, the criticality hazard associated with larger quantities of plutonium may be severe and must be carefully controlled to avoid disastrous experiences.^(9,46)

In summary, the hazards accompanying the handling of plutonium and its compounds which require the restrictions in handling stem from several sources. The principal restrictions arise from the intense alpha activity with its relatively short range and highly destructive effects in biological tissue. In addition, the neutron flux arising from the (α, n) reaction with light element constituents may be severe. Finally, the gamma flux accompanying the decay of plutonium and criticality considerations may be of importance.

II. GENERAL APPROACH TO PLUTONIUM HANDLING

Much has been written around the general philosophy applied to the handling of plutonium.^c Often as many approaches exist to some of the problems as there are workers in the field. It may be useful, therefore, to attempt to outline an agreeable basis of discussion from which an approach to the handling problem may be derived logically rather than to generate a set of arbitrary statements which at the outset will be a subject of debate.

One of the possible ways in which a sound approach to the handling problem can be derived is to consider the risk involved in a particular operation or program. The risk may in turn be evaluated by a consideration of a number of balancing factors. Important among these factors are:

^aBased on $10 \text{ n}/10^6 \alpha$ for F. The calculated value is higher than observed. See HW-55540, UCRL-3839.

^bLiterature on the handling of plutonium samples connected with high beta-gamma activity has been published. See for example, references 13, 28, 40.

^cSee for example, reference 1. p. 17, reference 15.

- (1) the quantities and forms of plutonium to be handled,
- (2) the urgency of the work to be performed,
- (3) the state of technology of the manipulations contemplated,
- (4) the alternative methods of carrying out the manipulations, each being evaluated in terms of some of the points listed below,
- (5) the safety features of the equipment and protection available to the workers,
- (6) the location or surroundings of the work area,
- (7) the consequences of unforeseen incidents,
- (8) the experience and character of personnel assigned to the work,
- (9) the economic aspect of the several alternative schemes, and
- (10) the available experience elsewhere in connection with similar or identical operations.

These factors cannot be placed in an unequivocally "correct" order, but can be placed into several categories. Those more easily evaluated on an objective basis include items 1, 5, 6, 9, and perhaps 3 and 10. The state of technology of the manipulations (No. 3) must be evaluated while keeping the restrictions of glove box work in mind if such restrictions are deemed necessary. Available experience (No. 10) may be sometimes difficult to ferret out and needs to be evaluated keeping differences in philosophy in mind.

More difficult to evaluate in the same manner are items (4) and (7). It is likely, for example, that several alternatives are available for carrying out the manipulations. To decide among them, however, requires the evaluation of such intangible factors as ease of work for the particular personnel, relative merit of the safety features of each procedure, etc.

More difficult still is the sensible evaluation of unforeseen incidents (No. 7). Although this appears at first to be self-contradictory, the procedure in question can be similar to evaluations carried out for reactor hazard studies or those made by insurance companies when evaluating insurance risks of all sorts, particularly industrial and atomic. It is mandatory in this instance that a sense of proportion be maintained. The magnitude of disasters evaluated should not exceed by a wide margin those which may normally be involved in a broad hazard evaluation of facilities larger and more extensive than that which is being examined.

Finally, evaluation of items (2) and (8) on an objective basis is probably not possible. The urgency of the work to be performed (No. 2) is clearly a relative matter and depends on the point of view of the evaluator. The experience of personnel is equally difficult to judge, particularly if a new operation is being considered. It is clear that the evaluation of a proposed operation within a glove box or the construction of a new facility can seldom be made a matter of routine. Such evaluations, however, are

normally made with surprising frequency, particularly in research facilities. A number of points may, in the light of the above discussion, be worth remembering.

1. The most conservative approach which minimizes the risk is not necessarily the optimum one.
2. The experience of others should be relied upon to a large extent but not the exclusion of reasonable modifications applicable to the circumstance. Equally important, procedures in use for some time should be readily abandoned if found to be unnecessary or inconvenient.
3. The experience and proven abilities of personnel assigned to the problem must be allowed to contribute substantially to both design and manipulation.
4. Although several reviews of plans and procedures are undoubtedly of value, the limitations of non-participating advice must be recognized. It is particularly true in this field where active manipulatory experience is of paramount importance.
5. Incidents must serve as object lessons for future conduct but not as opportunities for sweeping but illogical changes. Procedures which have lead to incidents should not a priori be discarded.

It is difficult to spell out in detail which of these considerations are applicable to a specific but undefined situation. Nevertheless, it is believed that an approach to the handlings of plutonium based on the above scheme should be realistic and workable. The use of this approach to evaluate new facilities as well as new manipulations in existing facilities is encouraged.

III. DESIGN OF ENCLOSURES

A. Introductory Comments

The confinement of activity represents the most widely used basic approach to the handling of plutonium and its compounds. Because of this fact, the design and construction of enclosures or boxes has received considerable attention. Unfortunately, little agreement exists among those experienced in the field of plutonium handling as to the most important or desirable design features of such enclosures.⁽⁴⁾ Considerable experience in a wide variety of operations is available at production facilities. Owing to the unique nature of the approach governing the handling of plutonium in production facilities, complete transfer of the design of

production boxes or trains to research facilities does not seem feasible. The differences in approach used in production facilities stem mainly from the evaluation of the risk factor as outlined in Section II.^a Because of these factors, only limited incorporation of procedures, design and construction of production facilities is possible for research equipment.

In addition, it is not feasible to discuss in detail all of the particular equipment used in research facilities. The discussion is of necessity vague on specific recommendations,^b and only in rare cases can one scheme or type of equipment be held more desirable than another. Opinions which are expressed below are, it is hoped, clearly labeled.

B. General Description of Glove Box Systems

Boxes and glove trains have been developed at several of the sites quite independently of each other for several reasons. The most important probably is the difference in the approach to the handling of plutonium. Thus, although samples of plutonium, its alloys and compounds are usually handled in completely closed facilities at some sites, open boxes, standard fume hoods or table top facilities have been used at other sites for work with plutonium. There are at least three topics of interest in this connection:

1. the design of the completely closed box,
2. the design of the open facility, and
3. the criteria which determine which of these types is to be used under specific circumstances.

The major part of the following discussion deals with Item 1.

1. Completely Closed Boxes

a. Glove boxes for research use have been arranged in several ways. Some sites favor boxes which are free standing in the laboratory.^(13,22,23,24,26,27) Access to the interior of the boxes may be through several of the vertical faces. Others have connected glove boxes into trains or glove box facilities. These are most frequently composed of boxes access to which is through only one face. The train of boxes may be arranged to minimize waste of laboratory space by either locating the boxes against the walls of the laboratory or by joining the boxes "back-to-back" in the interior of a laboratory. Combination of these two arrangements is popular where

^a See for example, reference 1, p. 9, paragraph 3; compare to reference 3, p. 3, Item B1, also reference 4.

^b For a listing of general design criteria of features affecting the fire hazard in glove boxes see reference 1, Appendix 3. See also references 4 and 15.

larger facilities are contained in large rooms. Finally, glove box trains have been assembled by positioning them against a wall adjacent to an access corridor. The latter represents a high contamination area and is entered by personnel clothed in protective suits.⁽²⁰⁾ Use of the access corridor is normally limited to decontaminating procedures, gross equipment changes and other operations not readily performed through the front of the boxes.

In a relatively small number of instances, double or triple walled boxes have been designed for high-level polonium chemistry and for operations deemed particularly hazardous. Completely enclosed boxes have also been placed within fume hoods for additional precaution. Special boxes have been built to house special equipment.^(8,21)

b. Glove boxes or trains of such boxes have been constructed of wood but more commonly of steel. Stainless steels have been used extensively but the use of mild steel suitably painted for corrosion protection has been found to be satisfactory and relatively inexpensive. Recently, the use of extruded aluminum framing has been tried with some success. In addition, whereas modular construction has in the past been based on a module composed of a complete box,^(4,16) recent developments have included the "erector-set" approach^(32,33) in which the frame of a box represents the module and the panels set into the frame are essentially custom sized. Enclosures are built around special equipment⁽²⁷⁾ rather than building equipment to fit into existing boxes. Rolled angle iron or "T" section steel⁽³³⁾ suitably welded together has also been suggested for glove box construction. It has been suggested that panels in large facilities be separated periodically by metal frame strips wide enough to constitute a fire break. Surface finish of glove boxes is governed by decontamination requirements and corrosion problems. Paints which are both resistant to chemical attack and fire are preferred. White and light green paints have been used.^a

At least two types of panels are required in glove boxes. Floors and some of the sides of the boxes are normally made of heavy gauge sheet metal or metal plate. The other type of panel must be transparent. The transparent panels fall into two types; those which must contain openings for glove ports and those which are unaltered and serve only for lighting and visibility. The latter generally do not represent a severe design and construction problem, but the need for heat resistance tends to favor tempered glass rather than safety glass which discolors under heat. The former because of reduction in structural strength brought about by the large glove port holes, have not yet been successfully made of glass. Current attempts at ANL to use tempered plate glass, laminated plate glass and other materials are only in the testing stage. Plexiglass 5009 is currently favored by several sites but has been rejected

^a See reference 47a, Appendix 1.

at ANL because of its low heat resistance (yield point). A limited discussion of window materials is available.⁽²⁾ Plexiglass or lucite has been used at ANL without incident but also has not been evaluated under fire or explosion conditions. In order to use glass, boxes are often designed so that the glove ports are set into a metal apron rising part way up the front.^a The rest of the front of the box is then covered by a sloping glass panel for visibility. This system unfortunately requires relatively elaborate metal work in the construction of the box and also reduces the visibility in the area inside the box immediately in front of the worker. It should be mentioned that minor modifications made on standard laboratory hoods (Type K, Blickman, for example) have resulted in a relatively inexpensive and very serviceable glove box, capable of containing experimental equipment in which gram quantities of powdered plutonium compounds have been handled without incident.^b

c. Panels attached to glove boxes are normally held in place by a positive system of clamps, clips, bolts, or similar mechanisms.⁽⁴⁾ Rubber gaskets are often employed to assure a tight seal. Thiokol is said to be superior to neoprene in very dry atmospheres.⁽⁹⁾ Recently, techniques used in the automotive industry have been applied at ANL with success to the problem of attaching transparent faces to the boxes. Heavy extruded rubber stripping holds the panels to the metal portion of the box. In addition, the transparent panel is cemented to the rubber and an expansion strip is forced into the rubber molding to increase the tension on both the transparent panel and the metal frame. Only the latter feature is new. This system has been tested quite thoroughly and is believed to represent a superior method of attaching transparent panels. It should be noted, however, that the permanent nature of the seal may be a disadvantage in those cases where panels may have to be changed frequently owing to surface deterioration by chemical or physical action.

Under normal operating conditions, it should not be necessary to require extreme tightness of the seals which hold the panels in boxes containing laboratory air at a slightly reduced pressure. This point is, however, a matter of some debate.^(9,13,32) Sealants for panel joints have included mastic, putty, adhesive tape, rubber, and others, all of which serve to restrict the air flow at the joints and to prevent particulates from diffusing to the outside of the enclosure. Automotive rubber stripping mentioned above when combined with a suitable cement produces a very tight seal proven suitable for inert atmosphere enclosures. Metal panels for bottom and sides of the boxes may be welded or clamped to the frame.

^a See for example, references 9, 12, 15.

^b See ANL-5875

d. The problems associated with the gloves and their attachment have been discussed in the literature.^(31,32,34) Currently, synthetic rubber gloves are probably the most frequently used, although some sites prefer the natural rubber glove. The permeability of the glove's material to moisture has been described in the literature.⁽³⁴⁾ Glove port sizes are, unfortunately, not standardized throughout the Atomic Energy Commission installations.^a At ANL, the 8-inch glove port has been strongly recommended as standard,⁽³¹⁾ but a large number of gloves 8 $\frac{5}{8}$ -inch size are already in use. In addition to this "standard" size, a 6-inch glove is available for use where space limitations exist.

An inexpensive 8-inch plastic glove port has been designed by CEN at ANL and is in use in several facilities.^b The main feature of this port aside from the low cost is the ease of mounting into panels. A single large hole cut into the panel is required for each glove port. Although the plastic glove port was designed for 3/8-inch panels, suitable spacers will permit mounting it into thinner material. Experience with this port has, to date, been satisfactory.

Glove ports have been designed with several different contours. Those currently in use at ANL have several grooves which accept rubber O-rings and/or metal clamping rings to hold the gloves in place. Glove ports with flat faces have also been used.⁽⁴⁾ At least two shapes of glove ports, oval and round, have been used. The latter are in use at ANL and elsewhere whereas the former is favored at LASL. A procedure for changing gloves without breaking the contamination barrier has been described.^c

e. One of the major sources of contamination in research facilities handling plutonium arises from transfer operations. In the course of moving active material into and out of the enclosures, the contamination barrier is changed from steel, glass and rubber to plastic pouches or laboratory air. Transfer of material within a set of boxes is generally less hazardous and is normally carried out by use of air locks, doors⁽¹⁵⁾ or conveyor-in-tunnel systems.⁽¹⁶⁾ Secondary containers are used. One of the most widely used methods of transfer into and out of the boxes is by means of plastic pouches.⁽³⁰⁾ At ANL and other sites, dielectric sealing devices are used to heat seal heavy (20 mil) polyvinylchloride plastic pouches.^d The sealed portion is separated from the rest of the pouch by

^a See also reference 47a.

^b See Sixth Hot Laboratory and Equipment Conference Proceedings, O.J.DuTemple, et al., p. 312. Also CEN Drawing No. CE-4108.

^c See for example reference 32. The method has been applied to inert atmosphere boxes.

^d See reference 29 for illustration of a small compact sealer used elsewhere. See also reference 30.

cutting the plastic along the center one of three seals. Pouches of glove port ($8\frac{5}{8}$ -inch) and larger diameter have been used. Thinner plastic pouches twisted to effect closure and cut across the twisted portion have been used extensively. The two twisted ends are taped to prevent spread of contamination. The latter technique has not been used at ANL but is preferred at LASL,^(4,16) and has simplicity and apparent success to recommend it.

Often the transfer of materials can be done by other than completely contamination-free means. Transfers of material into and out of glove boxes have been carried out by use of special cans or boxes attached to the underside of the glove boxes by lugs, press fit, gaskets, or similar mechanisms. This technique has been employed extensively at LASL and is described in the literature.⁽¹⁵⁾ Finally, under many conditions it is possible to remove contaminated material from a box train through an open faced hood. In this operation, a secondary container such as an ice cream carton is loaded in such a way as to have no contamination on the outside of the carton. This operation requires care, several glove changes and extensive monitoring.

f. Several types of ventilation systems are used in plutonium facilities. Systems using untreated air or a "once-through" basis are common. These draw air from the laboratory directly into the boxes. Inlet filters used in these systems serve to avoid diffusion of contamination out of the boxes and also remove particulate matter from the air.^(12,17) For these reasons inlet filters are normally required. Exhaust air is drawn through a set of filters prior to discharge into the atmosphere. Prefilters installed ahead of the final absolute filter bank are sometimes used, since such prefilters prolong the life of the final filter system and reduce the contamination in the duct work to a large extent.

Under many circumstances, normal laboratory air is not suitable for use in glove boxes. Drying of the air, mixing of air with an inert gas or using a completely inert atmosphere has been employed in such cases. Completely inert atmosphere boxes normally require a closed, circulating type of ventilation system. Facilities for drying or treating the atmosphere and for adjusting to rapid changes in pressure brought about by insertion or removal of hands into the gloves are provided.⁽¹⁷⁾ In addition, sensitive alarms coupled to the atmosphere purifying system are normally required to indicate leaks or glove failure. Systems using mixtures of dry air and an inert gas (example -30% He-dry air)⁽¹²⁾ are sometimes of the closed, circulating type,⁽²⁰⁾ but more frequently operate on a single pass basis.⁽¹⁾ Finally, static atmosphere systems have been employed in small glove box systems. The choice of atmosphere to be used is primarily dependent on the type of sample to be handled and the manipulations to be performed. A large part of chemical research probably could be done in "once-through" dry air boxes.

Considerable disagreement exists among sites as to the magnitude and direction of the air flow requirements in glove boxes, and it is not possible to make statements on this matter without arousing arguments.^a It seems reasonable, however, to require only sufficient air flow in closed boxes for heat removal and purity requirement (in case of inert atmosphere boxes) purposes while maintaining a suitable static pressure difference (0.2 - 0.8 inch of water). In addition, sufficient controlled capacity should be available to obtain a high flow of air through any leaks which develop suddenly, such as ruptured gloves or broken panels. Boxes having inlet filters and operating on a once-through laboratory air system are difficult to protect in this respect but the pressure drop across the inlet filter assures some flow of air through a torn glove opening. Manually operated multispeed exhaust systems are in use at some sites as boosters to the ventilating system of the room in which the plutonium facility is located. The air from these systems flows directly into a (sometimes separate) exhaust plenum. The latter is covered by a perforated steel plate to keep burning particles from entering the plenum in case of a fire in the room.⁽⁹⁾

g. The services required in glove boxes are normally not an integral part of the facility since limitations on their presence and use are necessary to avoid incidents.^b For example, the use of natural gas in glove boxes is discouraged primarily because of the explosive mixtures which may be formed without early detection. Drains from glove box facilities are always discharged to some sort of a retention tank system since permissible levels of plutonium in discharged water are very low. The use of water for cooling purposes is carefully controlled to avoid flooding the box and thus causing the spread of contamination.

Distribution of electric outlets can be built into glove box facilities if the need for explosion-proof outlets can be anticipated. The latter type of outlet seems to be preferred. It has been suggested that all services excluding electric outlets be introduced into glove boxes through operating faces by use of plugs or quick-disconnect fittings. Free standing boxes such as used by AERE connect all services and the exhaust into the boxes from overhead lines.⁽²⁶⁾ In the case of glove box systems for research purposes employing a laboratory air atmosphere introduction of services through the front has been found to be flexible and safe.

^a See for example reference 4, part 2a; references 12, 15, 16, 17 (The choice between metallic or plastic exhaust ducts is equally difficult.)

^b This point is one of debate. Although almost all of the published information on glove box design lists, in order, "...gas, electricity, water, compressed air, etc..." very little, if any, mention is made of the use of gas, for example, within the boxes. It appears that, at least in some instances, the inclusion of the whole gamut of services is based wholly on historical connections to fume hoods and bench top facilities.

2. Open Boxes

Open faced enclosures have been used extensively for plutonium chemistry. Such enclosures contain no glove ports but rather have an opening along the front normally about 7 inches high. Suitably protected hands are inserted through this slit which extends the full width of the box. Small removable plastic panels are located in the opening to reduce the amount of open area and the ventilation system is adjusted to give an adequate air flow through the opening. In some cases, the plastic panels contain two holes for insertion of hands. Such boxes are normally used for solution chemistry and some restriction is usually placed on the concentrations of plutonium permitted in the solutions and on the kind of operations to be conducted. The successful use of open boxes for plutonium handling requires more than the usual attention to a number of factors. Since no barrier exists between the contaminated area and the laboratory, the open box is the one most likely to lead to contamination. Transfer of materials out of the open box is normally minimized but can be done successfully with care. Continuous attention to monitoring of the area and checking of hands appears to be necessary. Those experienced in this type of operation are agreed that one of the most important ways to reduce contamination is by vigorous enforcement of good housekeeping rules within the open boxes. Ventilation must supply sufficient air velocity to avoid excessive outgoing eddy currents when moving across the open front and at the same time minimize turbulence of air within the box. Open faced boxes are used frequently for analytical chemistry and low-level routine work. The advantage of freedom of movement and absence of heavy rubber gloves is evident. The disadvantage of the open box arises from the lack of contamination barrier and the restriction on the kind and scope of operations which may be safely performed.

In addition to open boxes as described above, certain operations have been carried out in "table-top" arrangements which have only a very limited ventilating system.⁽¹⁵⁾ Since such arrangements are very close to bench top operation, the risk level is still higher than that for the open box. Successful operation of a "table-top" facility must rely on operator experience and care, relatively low levels of plutonium and meticulous attention to good housekeeping.

3. It is not logical to derive a set of regulations which permit a generalized, unqualified choice between the two types of facilities described above. It is commonly agreed that powdered samples of plutonium, its alloys or compounds are to be handled only in completely closed systems.^a Highly concentrated solutions of plutonium also are frequently handled only in closed systems.^b There is, however, sharp disagreement

^a See for example, references 12, 13, 18.

^b Reference 24 draws the line at about 100 mc Pu.

among experienced workers as to how much plutonium and in what concentrations it can be safely handled in open boxes. Clearly, the degree of confinement depends on evaluation of the risk as outlined in Section II. In this instance as in many others probably the most important single factor in evaluating this risk is operator experience and training. Data on the permitted plutonium concentrations are probably available from other sites, but it is believed that each facility (with its operators and supervisory personnel) represents a separate problem.

IV. SPECIAL PROCEDURES AND PROBLEMS

In the course of research on plutonium and its compounds, a large number of different types of operations have been performed under conditions not normally encountered.⁽¹²⁾ Some of these, pertinent to the work of the group and of the division, are briefly described in the following sections. In addition, certain problems peculiar to plutonium work are also discussed.

A. The handling of elemental fluorine is normally considered a reasonably hazardous operation, requiring considerable attention to the maintenance and design of equipment and to practices of good housekeeping. Elemental fluorine in combination with appreciable quantities of plutonium have been successfully manipulated by several workers.^a Several points of interest should be mentioned in this connection. Either large volumes or high pressures of fluorine should be avoided if possible. Where such extremes are necessary, adequate thought and precautions should be employed. Most materials of construction used in glove boxes are not fluorine resistant and several of the materials relied upon for contamination barrier (rubber, plexiglass, plastic pouches, etc.) will fail when directly exposed to fluorine. Fragile equipment such as pressure transmitters should not be allowed to fail to the exterior of the glove box system. In general, fluorine can be safely handled within glove boxes as the gas at relatively modest pressures. Valves regulating the flow of gases can be arranged external to the glove box. Disposal of fluorine to the glove box atmosphere is believed not desirable. Limestone traps vented to the glove box have proven satisfactory for the disposal of elemental fluorine. Equipment should be assembled with particular attention to the absence of organic materials or unpretreated metallic surfaces. Finally, dummy experiments using a stand-in for plutonium should be required not only in this case but for all untried operations.

^a See numerous papers on plutonium hexafluoride chemistry. See also ANL-5875 and reference 44.

B. Samples of solids submitted for X-ray diffraction analysis must be contained in capillaries which are both of suitably small diameter and show no external contamination.^a Preference has been expressed in favor of quartz capillaries of approximately 0.1 mm I.D. Experience has shown that such capillaries can be drawn from standard quartz tubing 8 - 9 mm O.D. Retention of an appreciable length of 8 - 9 mm tubing connected to the capillary makes holding and filling much easier. In addition, covering the capillary with a lusteroid test tube pressed against the shoulder formed by drawing the larger diameter quartz down to 0.1 mm will protect the exterior of the capillary against breakage and most contamination. Normally, considerable effort needs to be expended grinding the sample since the diameter of the capillaries is quite small. If pieces of material are stuck in the capillary, glass fibers made by drawing Pyrex rod can be used as drills and ram rods in some instances. When loaded, quartz capillaries can be sealed by an oxygen-gas flame. Considerable caution is needed in this operation. One of the methods used in sealing off capillaries is first to separate the large tubing from the main part of the capillary by sealing it off at the shoulder and then to seal the capillary at the desired length. Both operations are best carried out in an open front box. Several glove changes are usually necessary since the exterior of part of the capillary is normally severely contaminated. Tight fitting surgeon's gloves must be worn. After sealing the capillary to the desired length, the sealed tip is examined with a high power magnifying glass. Inclusions of plutonium in the sealed bead are normally not satisfactory since activity can be detected even though the plutonium is covered by glass.^b In that event, it is not possible to distinguish readily between fixed and movable contamination. Finally, the sealed capillary is decontaminated from adhering solids by dipping in various aqueous solutions. Finished capillaries are handled only in glass secondary containers and carried by storing the glass secondary in a beaker or similar container. Standard cameras have been modified and "remote" handling equipment has been designed which minimizes the hazard of breaking a capillary while loading it into the camera.^c

C. Considerable work involving optical microscopy and metallurgy of plutonium-containing systems has been done.^(9,15,19) Several references are available on techniques applicable here.

D. Problems of waste disposal⁽²³⁾ of plutonium are more severe than the corresponding problems for uranium. Disposal of plutonium-containing waste required attention to accountability requirements, health and criticality hazards and physical properties of certain forms of plutonium. At ANL, facilities for the recovery or reclamation of high-level

^a See also references 42 and 22.

^b Range of 5 Mev alpha in glass is approximately 0.02 mm.

^c D. S. Flikkema, ANL Chemical Engineering Division, private communication

plutonium-containing wastes are not available and hence all high-level wastes are shipped off site or stored. Procedures are available for the disposal of low-level aqueous waste. Concentrated aqueous solutions of plutonium produce gas due to the radiolysis of water. Concentrations of plutonium of perhaps 50 to 100 g/l probably require a venting system to avoid pressure build-up. Solid wastes which are chemically inactive are normally bagged and sent to a reclamation facility. Certain solids such as metal turnings or some plutonium alloys must be packaged carefully to avoid spontaneous combustion. Procedures are available in the literature to cover these cases.

E. Storage of plutonium-containing materials in glove boxes is normally held to a minimum. Storage areas are usually designed to be fireproof and critically safe even when flooded with water. Samples are usually bagged and always stored in secondary containers. The choice of materials for storage of the metal must be made carefully. It is recommended that plutonium metal be stored in aluminum rather than stainless steel cans since the heat conductivity of the latter is relatively low and plutonium is readily alloyed with iron.⁽¹²⁾ The need for this precaution in laboratory work should be evaluated.

F. Vacuum systems have been incorporated into glove boxes for research use. Evaluation of the risk of such facilities has led to several different approaches.^(13,20) Some sites have been quite hesitant in permitting glass vacuum lines in glove boxes. Much of the polonium chemistry carried out by Bagnall and associates on the other hand, was done in glass equipment enclosed in double-walled glove boxes.⁽¹⁴⁾ Glass blowing within a glove box facility requires more than the normal amount of skill and patience. Metal vacuum equipment has been used in glove boxes. Flare fittings, gasket closures and high-pressure connections have been successfully employed. Some sites require vacuum pumps to be either enclosed in the main glove box system or be enclosed in a separate box. Other sites limit requirements to venting of mechanical pumps into the glove box ventilation system. Experience has shown that the standard oil diffusion pump acts as a very efficient trap for plutonium.⁽¹³⁾ Further, limestone traps normally used in the disposal of experimental quantities of fluorine are an adequate trap for plutonium but will pass sufficient material to detect. Hence, such traps must be vented to the glove box system. Plutonium hexafluoride has been handled in metal vacuum line equipment located outside of glove box facilities, but care was required to avoid the spread of contamination. Vacuum gages used in this work are normally located for easy replacement. The repair of vacuum gages is normally not attempted. Introduction of coolants such as wet and dry ice into glove box facilities usually requires a bagging operation. Liquid nitrogen can be introduced into glove box facilities operating on laboratory air, through flexible hose by pressure applied to the external dewar. No check valve system appears to be necessary for this operation. Inert sides of pressure

transmitters represent a potential exit route for contamination and should be vented into the boxes. Finally, the pumping system of a vacuum apparatus should be so arranged that there is no possibility of evacuating the whole interior of a glove box facility constructed to withstand a vacuum of only a few inches of water.

G. Miscellaneous

A number of unique facilities have been described in the literature. These facilities incorporate many features too detailed to enumerate. A solvent extraction column system and plutonium casting equipment are described by Maraman.⁽¹⁶⁾ A complete analytical facility is described by Metz.⁽¹⁵⁾ Wiek and Thomas describe a metallurgical facility⁽¹⁷⁾ as do Shuck and Mayfield.⁽³⁷⁾ Cramer and Schonfeld report on the details involved in the metallography of plutonium.⁽¹⁹⁾ A general description of British facilities and operating principles is given by Lord and Waldron.⁽²⁰⁾ Several novel techniques applicable to glove box work are described by Bagnall and Spragg⁽²³⁾ and by Glanville and co-workers.⁽³⁵⁾ High-temperature fused salt research is described by Barton and co-workers.⁽³⁸⁾ Spectrographic analyses are described by Birks.⁽⁴³⁾

V. SAFETY, CONTROL OF ACTIVITY, AND EMERGENCY PROCEDURES

A. Considerable material has been written on the control and prevention of fires within glove box facilities.^(2,5,6,7) Some of the pertinent points are summarized below:

1. Metallic samples of plutonium are normally quite reactive and in some instances pyrophoric. Storage of such forms of plutonium is usually limited to inert atmosphere fire-proof containers. Turnings and metal chips may ignite in air by friction contact with other turnings or chips. Plutonium reacts with water in the absence of oxygen to form an extremely reactive hydride which will catch fire on subsequent exposure to air.⁽¹²⁾ The choice of atmospheres must be judicious.^a
2. Some workers experienced in the field believe that confinement of fires within glove box systems is preferred to attempting to put the fire out. It has been stated that plutonium fires in an air atmosphere cannot be extinguished. Magnesium oxide sand and asbestos blankets are sometimes used to control the spread of plutonium

^aSee also references 4, 36.

fires.⁽¹⁾ Use of coolants such as water or CO₂ from a fire extinguisher or the use of large copper blocks⁽¹²⁾ is recommended for cooling of surfaces in contact or in the vicinity of the fire. Use of water applied to exterior surfaces of the plastic panels is recommended by some as being sufficient to prevent damage to the plastic. There seems to be agreement that the use of water within glove boxes for fire control is to be used only as the ultimate alternative. Sprinkler systems are installed in some facilities to protect the filter systems and plenums.⁽¹⁾ Sprinkler systems are normally never installed to operate within a plutonium facility.

Carbon dioxide extinguishers operated from the outside of the glove box are preferred in some installations. Others use several types of extinguishers in combination such as CO₂, dry chemical and Met-L-X types. Difficulty has been experienced in the use of CO₂ within boxes because of (a) the ventilation system removing CO₂ as fast as it is being introduced thus negating its effect and (b) a closed system suddenly flooded with large volumes of gas leading to rupture of gloves or panels. Manually operated or automatic regulators installed in appropriate places in the ventilating system are believed adequate to overcome the first objection. The second point unfortunately does not seem to have a ready solution in small boxes except to change the procedure of fire retardation.

3. All are agreed that a carefully designed and often practiced fire drill is necessary. Considerable effort has been expended by other sites to not only develop such a routine but also educate the personnel. The written procedures resulting from such programs are available in some cases.
4. Much can be done to prevent fires in glove boxes.^a As mentioned earlier, fireproof construction involving glass or metal panels, a minimum of flammable paint, compartmented boxes, the use of fireproof or fire resistant filters, etc., have been proposed and used. Metal glove port covers attached to the inside of the plastic glove port have been developed by CEN. Others have used glove port covers clamped to the outside of the port. Good housekeeping is an obvious method of reducing the fire hazard. Some sort of fire detection equipment is in use at most sites. Some of these detect the rate of change of temperature, some are

^aSee discussion in reference 24 for example.

set for a fixed temperature, and others are capable of detecting smoke.⁽¹⁾ In some instances these devices are connected to fire extinguishing equipment but more frequently they actuate alarms,⁽¹⁾ both local and general. Although usually not a major point in small-scale operations, the construction of the building or the laboratory in which plutonium facilities are to be constructed should be kept in mind when evaluating fire hazards and overall risk.

B. Certain regulations enforced by various groups pertain to materials located within the glove boxes. Many such regulations exist and a number of them are abstracted below.

1. Under normal conditions flammable solvents are not permitted in glove boxes. Coolants used in machining and metallurgical operations are chosen carefully. The use of highly volatile solvents has led to unfortunate incidents⁽⁵⁾ and should be avoided. Where solvents are necessary, a scrubbed recirculating ventilation system is often used.⁽¹⁾ Rags and other types of materials likely to be the cause of fires are restricted.
2. Perchloric acid, when used in analytical facilities is sometimes confined to glove boxes equipped with ventilation scrubbers to avoid formation of explosive deposits in the ventilation duct work. Not all sites agree on the necessity for such precautions.
3. The use of sharp objects such as scissors, knives, razor blades, etc., is minimized wherever possible.⁽¹⁶⁾ When scissors are necessary, it is feasible to remove the points by grinding the tips. Broken glass is normally removed as soon as it is detected. Cloth or leather gloves or liberal use of tongs and tweezers are encouraged when sharp metallic objects are handled.
4. Control of the quantity of plutonium in a glove box facility is governed by criticality considerations. These are probably unique for each facility and except to mention the point no further discussion seems warranted.
5. Some sites do not permit small vacuum cleaners to be used within a glove box. The use of vacuum devices connected to suitable filters has proven to be very useful for particulate matter.⁽²⁵⁾

C. The control and detection of contamination has been discussed in several places above.^a A number of additional points may be of interest.

1. Some sort of protective clothing is normally required for those working with plutonium. Open box work requires the use of rubber gloves and usually laboratory coats which are taped at the sleeves. Some sites require the use of this type of clothing also for work in closed glove boxes. Washable booties are in use in several places as is the procedure of completely changing clothes before entering the plutonium area. Several types of respiratory equipment are available when circumstances require their use. The self-contained type using tank breathing air is preferred under high-risk situations. As a rule, self-contained respirators, protective clothing and gloves are required for new and untried operations irrespective of their simplicity.
2. Procedures have been developed which were designed to detect contamination at the earliest possible moment. No apparent uniformity of detail exists among the sites. It is a requirement of some sites that hands must be checked after every withdrawal from the glove box system.⁽¹⁷⁾ At some sites, counting equipment for this purpose is mounted on the glove box. Frequent checking of feet is normally required. Some sites have installed continuous air monitoring equipment⁽³⁹⁾ while others check the air only infrequently. Procedures for checking personnel by urine counts, nose swabs, etc., vary among the several sites. Routines followed by Health Physics groups differ among the sites,^(3,48) as do the number of Health Physics personnel assigned to a group of workers. Almost all sites enforce some sort of separation between the office area and the laboratories or production lines. Eating, drinking, and smoking in the laboratory is prohibited in most, but not all, installations.
3. Alpha monitors using a very thin nylon, rubber, or coated mylar membrane and air or a counting gas are normally in use for surface contamination detection. These may be battery operated and portable or AC operated. Permissible limits of fixed contamination vary from several M^b to fractions of an M. Removable contamination as determined by surface smears is usually required to be less than detectable.^c

^aSee also references 10b, 19, 23.

^b1 M = 10^3 d (min)(100 sq cm)

^cDetectable limits vary, but on the "average" are about 0.01 M

Air sampling often uses filter paper through which air has been drawn.⁽¹⁸⁾ The paper is counted, allowing for the decay of radon daughter products. Two types of air count systems are in use. In one type individual units made of modified vacuum cleaners pull a metered amount of air through a filter paper. Another type uses a system of pipes connected to a common pumping system. The pipes terminate in several laboratories and are covered with small circles of filter paper. Periodically, the filter paper is removed and counted.

VI. SUMMARY

Many of the features of plutonium facilities have been covered and a number of them have been omitted. A great variety of safety equipment is available, together with trained personnel to operate it. All of these devices, however, do not assure a contamination-free operation. Basically, the careful design of enclosures, experimental equipment and procedures when handled by trained personnel represents the only approach to the problem of plutonium handling.

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